

# INVESTIGATIONS OF DEFECTS IN PROTECTIVE FILMS WITH OPTICAL AND ELECTROCHEMICAL FUNCTIONAL NSOM TECHNIQUES

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*Functionalized* near-field scanning optical microscopy (*f*-NSOM) which allows concurrent scanning electrochemical microscopy (SECM) and topography measurements has been employed to characterize the physical properties of defects and flaws in protective films on metals. The analysis of these surfaces is a multistep process, where areas of interest are initially identified using dark-field microscopy which highlights areas with topographical irregularities in and on the surface, thereby allowing fast scanning and cursory inspection of large surfaces to locate areas where further investigation is warranted.

Many other techniques besides optical dark-field microscopy offer similar or better information about the topography of the surface, such as AFM and SEM are also available. While AFM is superior in quantifying the topographical features, it offers little information regarding the reactivity of the surface in and around the anomalies. However, dark-field optical microscopy is very nice for visually scanning the surface because it is fast and non-destructive, yet small features may be reliably detected.

The important question still remains once the interesting sites have been identified: Is there a reactive metal surface that is exposed through pinholes in protective overlayers? This question is particularly interesting with regards to magnetic disk-drive manufacture as ever thinner diamond-like carbon (DLC) films are utilized to protect sensitive surfaces in data storage devices from wear and corrosion, as information density continues to increase. This continuing evolution of technology drives the necessity of developing better techniques for analyzing the quality of such protective films.

Two approaches to characterizing holes in protected films are used: In one, reactive surfaces are decorated with small molecules that enhance optical detection by changing the reflectance, absorption or photoluminescence properties of the surface. One requirement for such sensor molecules is that they must accumulate preferentially at sites not covered by the protective film. Detection by AFM and SEM is also enhanced in this approach, as the topography of the decorated surface is different from that of the coated surrounding surface. In the second approach, *f*-NSOM measurements that provide quantitative information about the topography and concurrent information about the conductive properties of the surface that is readily correlated to the topography images. This second approach is advantageous to the first one in that it is less intrusive, since it does not alter the chemical properties of the surface in mapping them out.

Our group has previously developed a *f*-NSOM, based on the shear-force feedback tuning fork

technique, for use in liquids.<sup>1</sup> Due to the versatility of this design, we have the ability to investigate samples in solution as well as in air. Thus, we are able to routinely obtain scanning electrochemistry microscopy (SECM) images as well as standard optical NSOM images in addition to the concurrently obtained topographical images.<sup>2-4</sup> These advantages over the commercially available systems allow us to perform SECM experiments where the tip is scanned at a constant distance of 0 to 30 nm over the surface,<sup>3</sup> while the potential of the tip and surface are carefully controlled, as is done in traditional SECM experiments.<sup>5</sup> Hence, this unique technique also offers specific advantages over the traditional SECM in that the artifacts stemming from changes in the sample-probe distance on uneven surfaces are diminished.

As we are continuously striving to improve the lateral resolution of the *f*-NSOM technique, new techniques for preparing highly reproducible tip geometries, such as focused ion beam milling (FIBM) are being employed. In order to demonstrate the state-of-the-art in *functionalized* scanning probe microscopy (SPM), a number of investigations of well-characterized substrates, such as holes created by FIBM on DLC films covering copper substrates, will be presented.

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## References

1. P. I. James, L. F. Garfias-Mesias, P. J. Moyer and W. H. Smyrl, *J. Electrochem. Soc.* **145**, L64, (1998).
2. L. F. Garfias-Mesias and W. H. Smyrl, *J. Electrochem. Soc.* **146**, 2495, (1999).
3. J. Kerimo, M. Büchler and W. H. Smyrl, *Optical Metrology*, G. A. Al-Jumaily, Ed. PV CR72, p. 232. SPIE Optical Engineering Press, Bellingham, WA (1999).
4. M. Büchler, J. Kerimo, F. Guillaume and W. H. Smyrl, *J. Electrochem. Soc.* **147**, 3691, (2000).
5. A. J. Bard, F.-R. Fan, D. T. Pierce, P. R. Unwin, D. O. Wipf and F. Zhou, *Science*, **254**, 197, (1991).